

GC-MS EXAMINATION OF METHANOLIC EXTRACT OF *Cirsium arvense* FLOWERS***Malik F. H. Ferdosi¹, Iqra Haider Khan², Arshad Javaid² and Muhammad F. A. Fardosi³**DOI: <https://doi.org/10.28941/pjwsr.v27i2.946>**ABSTRACT**

Cirsium arvense (L.) Scop., commonly known as creeping thistle, is a weed of Asteraceae. This study was undertaken to explore various phytoconstituents present in flower of this weed. To achieve this goal, the dried flowers of this weed were soaked in methanol for one week and filtered. This methanolic extract was subjected to GC-MS analysis and 7 compounds were identified. These included olean-12-en-3-ol, acetate, (3 β)- (63.87%), lanosta-8,24-dien-3-ol, acetate, (3 β)- (12.12%), β -amyrin (6.19%), γ -sitosterol (6.09%), α -amyrin (5.24%), stigmasterol (3.29%) and carbonic acid, 2-ethylhexyl heptadecyl ester (3.16%). Literature survey showed that these compounds possess anti-inflammatory, antimicrobial, antidiabetic, antioxidant and/or anticancer activities.

Keywords: Creeping thistle, Flower extract, GC-MS analysis, Phytochemicals.**Citation:** Ferdosi, M.F.H., I.H. Khan, A. Javaid, M.F.A. Fardosi. 2021. GC-MS Examination of Methanolic Extract of *Cirsium arvense* Flower. Pak. J. Weed Sci. Res., 27 (2):173-180.

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INTRODUCTION

Natural compounds isolated from plants have recently become of great interest because of their variable biological applications (Lichota and Gwozdziński, 2018; Javed *et al.*, 2021). These are considered as the affluent bio-resource of food supplements, nutraceuticals, pharmaceutical intermediates as well as folk, traditional and modern medicine systems (Jamiołkowska, 2020; Khan and Javaid, 2020a). It has been estimated that 15–29% of higher plant species extract contain synergistic effects and polypharmacological applications to treat human as well as plant diseases (Yuan *et al.*, 2017). The identification and purification of active principles produced by plants are of supreme importance to ensure their efficacy and safety in pharmaceuticals (Jiménez-Reyes *et al.*, 2019). The determination of phytoconstituents is largely dependent on the solvent type, which is used for extraction. So far, methanol is one of the most commonly used solvents to investigate the biological activities from plant extracts (Sauerschnig *et al.*, 2018; Banaras *et al.*, 2021). Moreover, dried or fresh plant materials exist in their biologically active forms and are used as a source of secondary components (Ingle *et al.*, 2017; Javaid *et al.*, 2018). Many scientists are working on the phytoconstituents and have attracted the attention of scientific communities towards the application of dried plant materials in agriculture and pharmacology (Ali *et al.*, 2020; Khan and Javaid, 2020a,b).

Cirsium arvense, family Asteraceae, is native to Western Asia, Europe, North Africa, eastern United States and Canada (Nobarinezhad *et al.*, 2020). It is very common in different parts of the province Punjab. It is a perennial persistent noxious weed plant that expands vigorously by horizontally grown roots and forms dense colonies (Verbeek and Kotanen, 2019). It spreads through wind or as a contaminant in seed crops in the temperate parts of the world.

It infests many habitats such as roadsides, cultivated fields, rangelands, pastures, embankments, lawns and railways (Carter and Lym, 2017). It is a source of tannins, triterpenes, sterols, phenolic acids, coumarins and flavonoids (Dehjurian *et al.*, 2017). It showed antifungal activity against *Macrophomina phaseolina* (Banaras *et al.*, 2017) and allelopathic potential against weeds and crop plants (Bajwa and Javaid, 1995; Akhtar *et al.*, 2001). It has medicinal uses for the treatment of metrorrhagia, epistaxis, syphilis eye infections, bleeding piles, skin sores gonorrhoea, leukaemia, peptic ulcer and tuberculosis (Amiri *et al.*, 2018). Information regarding phytochemical analysis of flower extract of this weed growing in Pakistan is lacking. Thus, the present study was carried out to explore the phytochemical profile and biological active constituents from the methanolic extract of *C. arvense* flowers.

MATERIALS AND METHODS

Preparation of methanolic extract

The full bloomed flowers of *C. arvense* were collected from Lahore, Pakistan. The plant species was identified by a botanist Dr. Arshad Javaid (also an author of this paper). The collected material was shifted to the lab to dry under shade conditions. For full drying, the specimen was put in an electric oven at 35 °C for 5 hours, grinded into powdered form and completely soaked in commercial grade methanol in closed media bottle for 15 days. Then the solvent was filtered by using double filter paper and 2 mL sample was taken into a glass vial for GC-MS analysis.

GC-MS analysis

The sample was analyzed by GC-MS for the identification of various components of methanolic flower extract of *C. arvense*. The spectrum of each compound was compared with the compounds in library and arranged in the ascending order of their retention times. The relative abundance was reported by using their peak areas. The structures of the identified natural compounds were drawn by using ChemDraw software. The set GC-MS conditions are given in Table 1.

Table 1: GC-MS conditions.

GC Program	
Column	DB-5MS (nonpolar), (30 m × 0.25 µm × 0.25 µm)
Equipment	Agilent Technologies (GC-7890B: MS-5977A)
Injection Temperature	250 °C
Carrier gas	Helium gas 1 mL min ⁻¹ , split less mode
Detector	Mass detector
Software	Mass Hunter
Sample injection	1 µL
Oven ramping	Initial 50 °C and then raised 10 °C per min up to 290 °C
Total GC run time	50 min
MS program	
Library used	NIST version 2017
Inlet line temperature	280 °C
Source temperature	230 °C
Quadrupole Temperature	150 °C
Mode:Scan (m/z)	50–500
Solvent delay	3 min
Ionization voltage	70 eV
Total MS run time	50 min

Literature survey

An online extensive survey of the relevant published scientific articles for finding out the bioactivity of identified natural components of methanolic flower extract of *C. arvense* was performed.

RESULTS AND DISCUSSION

Details of the seven natural compounds identified in this study are given in Table 2 while their properties are presented in Table 3. The predominant compound in this study was olean-12-en-3-ol, acetate, (3β)- with 63.87% peak area. The second most abundant compound was lanosta-8,24-dien-3-ol, acetate, (3β)- with 12.12% peak area. Other compounds identified in this study were β-amyrin (6.19%), γ-sitosterol (6.09%), α-amyrin (5.24%), stigmasterol (3.29%) and carbonic acid, 2-ethylhexyl heptadecyl ester (3.16%). Structures of these compounds are presented in Fig. 1.

The most abundant compound in this study was olean-12-en-3-ol, acetate, (3β)-. It is also known as 3-β-acetoxylean-12-ene or β-amyrin 3-acetate. Previously, this compound has been identified in *Cornus macrophylla* as the fifth most abundant compound (Akbar *et al.*, 2020). It has also been reported in *Sambucus chinese* (Yang and Lin, 2004).

Fruit of *Manilkara subsericea* is a rich source of this substance as 72.81% of its hexanic extract was composed of mixture of α- and β-amyrin 3-acetate (Fernandes *et al.*, 2013). Both of these isomers possess anti-inflammatory activity (Akihisa *et al.*, 2010). In addition, β-amyrin 3-acetate also showed antibacterial activity by inhibiting the growth of *Staphylococcus aureus* (Hichri *et al.*, 2003).

α-Amyrin and β-amyrin were found as moderately abundant compounds in the present study. The amyrins are present in three forms namely α-, β- and δ-amyrin, each having a pentacyclic triterpenol and formula C₃₀H₅₀O. They are extensively found in nature and have been identified in a number of plant species including *Myrcianthes pungens* and *Strobilanthes callosus* (Singh *et al.*, 2002; Cardoso *et al.*, 2020). The α- and β-amyrin from *Protium heptaphyllum* have many pharmacological actions in many systems, for example immunological system and gastrointestinal tract (Nogueira *et al.*, 2019). These are also known to have antioxidant, antimicrobial and anti-inflammatory activities (Singh *et al.*, 2002; Cardoso *et al.*, 2020). These compounds can be used as therapeutic

agents to treat periodontitis and gingivitis because they adjust acute periodontal inflammation by lowering oxidative stress and neutrophils infiltration, and by producing pro-inflammatory cytokine TNF- α (Pinto *et al.*, 2008).

Stigmasterol was identified as a minor compound in this study. It is an unsaturated sterol that has been isolated from a number of medicinal plants. It involves in the formation of various hormones such as estrogens, progesterone, corticoids and androgens. Moreover, its various derivatives such as cyasterone, spinasterol, stigmasterol glucoside, fucosterol, fucosterol epoxide,

etc. have been identified and their pharmacological aspects have been observed (Kaur *et al.*, 2011). Zeb *et al.* (2017) isolated this compound from roots of *Indigofera heterantha* and reported its anti-inflammatory activity. Wang *et al.* (2017) identified this compound in soybean oil and reported its anti-diabetic activity. Stigmasterol isolated from *Inula britannica* showed lethal effect against the mite *Tetranychus cinnabarinus* (Cheng *et al.*, 2012). γ -Sitosterol was a moderately abundant compound in this study and is known to possess anticancer and antidiabetic potential (Balamurugan *et al.*, 2011; Sundarraj *et al.*, 2012).

Table 1: Compounds in methanolic flower extract of *Cirsium arvense*.

Sr. No.	Names of compounds	Molecular formula	Molecular weight	Retention time (min)	Peak area (%)
1	Carbonic acid, 2-ethylhexyl heptadecyl ester	C ₂₇ H ₅₄ O ₃	426.7	15.879	3.16
2	Stigmasterol	C ₂₉ H ₄₈ O	412.7	45.874	3.29
3	γ -Sitosterol	C ₂₉ H ₅₀ O	414.70	47.463	6.09
4	β -Amyrin	C ₃₀ H ₅₀ O	426.7	48.244	6.19
5	α -Amyrin	C ₃₀ H ₅₀ O	426.7	49.463	5.24
6	Lanosta-8,24-dien-3-ol, acetate, (3 β)-	C ₃₂ H ₅₂ O ₂	468.75	49.961	12.12
7	Olean-12-en-3-ol, acetate, (3 β)-	C ₃₂ H ₅₂ O ₂	468.75	50.763	63.87

Table 2: Bioactivity of components of methanolic flower extract of *Cirsium arvense*.

Sr. No.	Names of compounds	Bioactivity	Reference
1	Carbonic acid, 2-ethylhexyl heptadecyl ester	-	-
2	Stigmasterol	Anti-inflammatory, anti-diabetic, acaricidal	Cheng <i>et al.</i> (2012); Wang <i>et al.</i> (2017); Zeb <i>et al.</i> (2017)
3	γ -Sitosterol	Antidiabetic, anticancer	Balamurugan <i>et al.</i> (2011); Sundarraj <i>et al.</i> (2012)
4	β -Amyrin	Antioxidant, anti-inflammatory, antimicrobial	Singh <i>et al.</i> (2002); Cardoso <i>et al.</i> (2020)
5	α -Amyrin	Anti-inflammatory, antioxidant, antimicrobial	Singh <i>et al.</i> (2002); Cardoso <i>et al.</i> (2020)
6	Lanosta-8,24-dien-3-ol, acetate, (3 β)-	-	-
7	Olean-12-en-3-ol, acetate, (3 β)-	Anti-inflammatory, antibacterial,	Akihisa <i>et al.</i> (2010); Hichri <i>et al.</i> (2003)

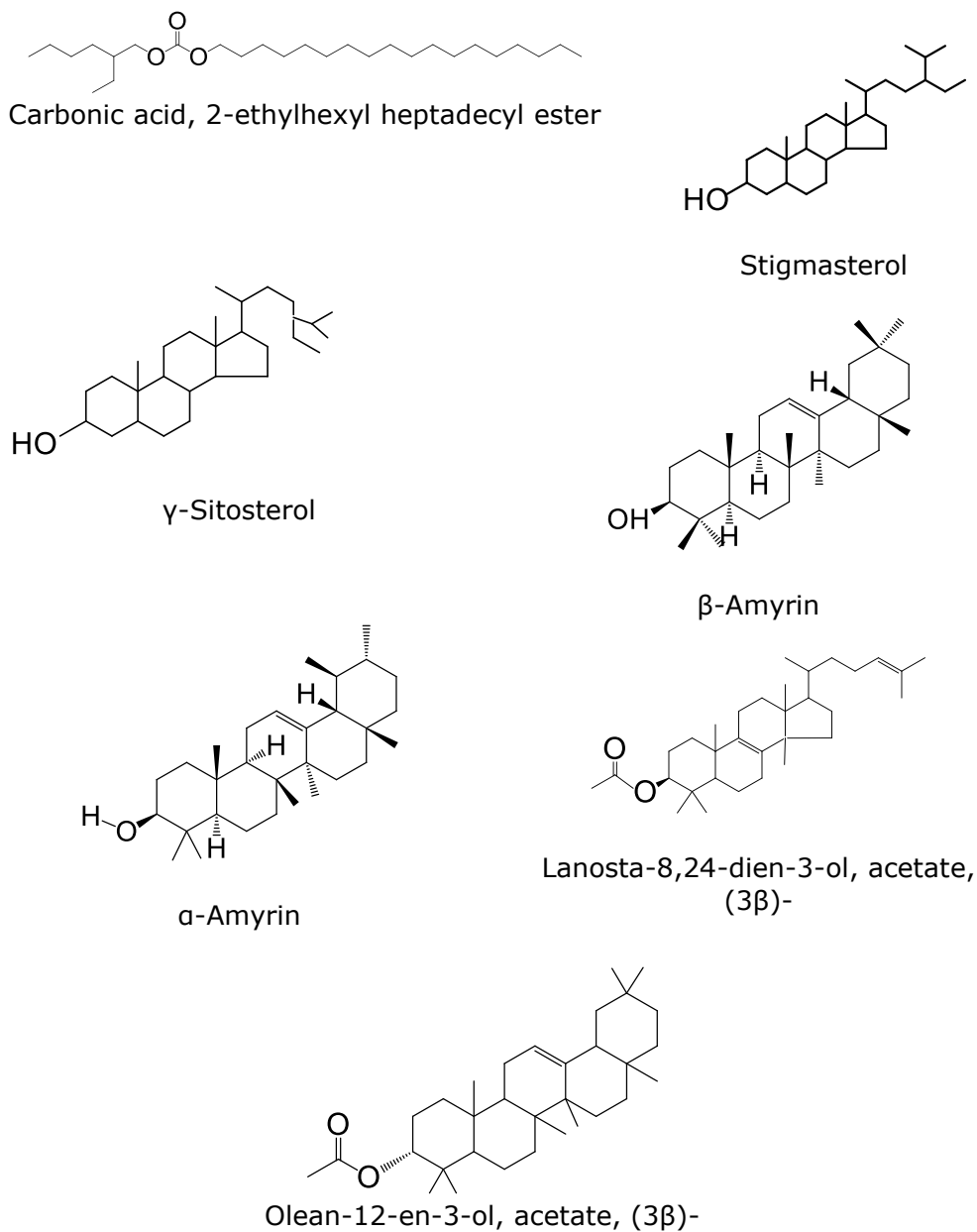


Fig. 1: Structures of compounds in methanolic extract of flowers of *Cirsium arvense*.

Conclusion

This study concludes that methanolic flower extract of *C. arvense* is a rich source of many pharmaceutically important compounds such as olean-12-

en-3-ol, acetate, (3 β)-; α and β -amyrin; γ -sitosterol and stigmasterol, having anti-inflammatory, antimicrobial, antidiabetic, antioxidant and/or anticancer properties.

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